IMP. BUR.
17 JUN 1920

UNITED STATES DEPARTMENT OF AGRICULTURE

BULLETIN No. 855

Contribution from the Bureau of Animal Industry
JOHN R. MOHLER, Chief

Washington, D. C. PROFESSIONAL PAPER May 6, 1920

SAPONIFIED CRESOL SOLUTIONS

By JACOB M. SCHAFFER, *Junior Chemist, Biochemic Division*

CONTENTS

	Page.		Page.
Properties of mixtures with rosin soap	2	Cost of materials used	4
Observations on speed of dilution	3	Summary	5

This paper describes a series of experiments undertaken with the object of preparing a saponified cresol solution which would be cheaper and at the same time no less effective as a disinfectant than those at present in use. Such a product should contain the usual 50 per cent cresol with the necessary quantity of soap to insure complete solubility in water and should meet the following requirements:

The product shall remain a homogeneous liquid when cooled to 32° F. It shall contain substantially no free oil, fatty acid, or excess alkali. It shall be readily soluble in cold distilled water; the solution shall be practically clear and shall contain no globules of undissolved oil or cresylic acid.[1]

By stating the qualifications of a good finished product the Bureau of Animal Industry has safeguarded itself and at the same time allowed the manufacturer to mix the necessary ingredients in the manner he finds most economical.

Disinfectants made with cresylic acid and rosin soap have been objected to because they become cloudy when diluted with water. In the case of a cresol-rosin soap solution diluted with water to a 3 per cent solution and exposed in a flask to the air, it has been found that the clouding is due to the hydrolysis of the rosin soap and the absorption of carbon dioxid from the air, the rosin being precipitated at first in finely divided particles. After a few days these particles commence to agglomerate and settle to the bottom; finally, after about two weeks, equilibrium is reached, the solution clears, and shows a good deal of rosin in a mass at the bottom of the

[1] U. S. Bureau of Animal Industry Order 263.

171564°—20

flask. Further dilution increases the hydrolysis and subsequently brings on additional clouding.

PROPERTIES OF MIXTURES WITH ROSIN SOAP

It was found that soaps of some vegetable oils, fatty acids, and fish oil (menhaden) when mixed with rosin soap had the property of retarding the clouding and precipitating of rosin in rosin-soap-cresol solutions. A number of different soaps were tried as ingredients of 50 per cent saponified cresol solutions. Those tried were rosin soap, soy-bean-oil soap, linseed-oil soap, oleic-acid soap, fish-oil soap, rosin-soy-bean-oil soap, rosin-linseed-oil soap, rosin-oleic-acid soap, and rosin-fish-oil soap. These soaps were used in proportions equivalent to 28 per cent linseed oil in the finished product.

Saponified cresol solutions were made with rosin soap, the sodium hydroxid varying from enough to saponify the rosin to an excess of 20 per cent. On dilution to a 3 per cent solution with distilled water the saponified cresol solution containing no excess alkali dissolved quickly and clouded after about 3 minutes; beyond 5 per cent excess alkali the solutions dissolved more slowly and clouded after about 3 minutes. Increasing the excess of alkali beyond 5 per cent has therefore no beneficial results, and at the same time this excess combines with the cresol, lowering the coefficient of the finished product.

Saponified cresol solutions were prepared by mixing rosin and soy-bean oil, saponifying the mixture with just enough sodium-hydroxid solution and then adding the cresol. These had the same properties as saponified cresol solutions containing varying proportions of rosin soap to soy-bean-oil soap prepared by mixing a saponified cresol solution containing soy-bean-oil soap and one containing rosin soap.

Analysis of the soy-bean oil saponified cresol solution:

Phenols, 53.5 per cent.
Fatty acids + unsaponifiable matter equivalent to 28 per cent oil.
Total alkali as sodium hydroxid, 4.0 per cent.
Water not determined.

Analysis of the rosin saponified cresol solution:

Phenols, 53.5 per cent.
Rosin acids (equivalent to 33.6 per cent rosin or 28 per cent oil), 30.2 per cent.
Total alkali as sodium hydroxid, 4.0 per cent.
Water not determined.

The table following gives the proportion of soy-bean-oil soap to rosin soap in the total soap present in these saponified cresol solutions. Under the heading "Remarks" are recorded the results of adding the saponified cresol solutions to distilled water at 20° C. to form 3 per cent solutions.

Soy-bean-oil soap.	Rosin soap.	Remarks.
Per cent.	*Per cent.*	
10	90	Shows faint clouding after 1½ hours.
20	80	Shows faint clouding after 2½ hours.
25	75	Clear after 5 hours; faint cloud at 24 hours.
30	70	Clear at 24 hours (sometimes faintly cloudy).
40	60	Faint cloud after 48 hours.
50	50	Clear at 7 days.
60	40	Clear after 7 days.
70	30	Do.
80	20	Do.
90	10	Do.

OBSERVATIONS ON SPEED OF DILUTION

All dilute clear and more quickly than the saponified cresol solution prepared from soy-bean oil only; those containing the larger proportion of soy-bean oil to rosin more nearly approach straight soy-bean oil in speed of dilution. None separate or solidify when held at 32° F. All are more viscous than the straight soy-bean oil preparation. Amounts of sodium hydroxid up to 20 per cent in excess of that theoretically required in similar preparations retard the speed of dilution and only to a slight extent the clouding.

Three per cent dilutions were made, using tap water instead of distilled water. The saponified cresol solution made with straight soy-bean-oil soap clouded immediately. The soy-bean oil 10 per cent, rosin 90 per cent, clouded after about one-half hour. The soy-bean oil 30 per cent, rosin 70 per cent, was clear at 5 hours but cloudy at 24 hours. Amounts of sodium hydroxid up to 20 per cent in excess of that theoretically required in preparations like these had little effect on their tendency to cloud.

A saponified cresol solution made by mixing a saponified cresol solution containing linseed-oil soap and the solution containing the rosin soap so as to contain a soap equivalent to linseed oil 25 per cent, rosin 75 per cent, behaves very much like that made with soy-bean oil 25 per cent, rosin 75 per cent.

A saponified cresol solution made by mixing a solution containing fish-oil (menhaden) soap and the solution containing the rosin soap so as to contain a soap equivalent to linseed oil 25 per cent, rosin 75 per cent, behaves very much like that made with soy-bean oil 25 per cent, rosin 75 per cent.

A saponified cresol solution containing 7 per cent by weight of soy-bean oil as soap and 25 per cent by weight of rosin (containing 90 per cent rosin acid) as soap will closely approximate a saponified cresol solution made by using a mixture of 25 per cent soy-bean-oil soap and 75 per cent rosin soap. The figures are convenient to use and a preparation made with this proportion of oil to rosin should be just as easy to manufacture as the saponified cresol solutions at pres-

ent in use. It may be easier, in fact, because the presence of rosin hastens the saponification of the oil.

The mean combining weight of rosin was taken as 346.[1]

Saponification value of 1 gram of linseed oil in terms of sodium hydroxid equals 0.137 gram.

$28 \times 0.137 =$ 3.83 grams sodium hydroxid needed to saponify 28 grams oil.

$3.83 \times \frac{346}{40} = 33.13$ grams rosin equivalent to 28 grams oil.

$33.13 \times \frac{3}{4} = 24.82$ per cent rosin (approximately 25 per cent).

This rosin-soy-bean-oil-cresol solution remains a homogeneous liquid when cooled to 32° F. It is readily soluble in distilled water. The freshly prepared 3 per cent solution is clear and remains clear for more than 1 hour. The rosin-linseed-oil-cresol solution, rosin-oleic-acid-cresol solution, rosin-fish-oil-cresol solution containing 25 parts by weight of rosin and 7 parts by weight of oil or equivalent in fatty acid have these properties also.

COST OF MATERIALS USED[2]

Sodium hydroxid, 76 per cent, 4 pounds, at $3.25 per 100 pounds	$0.13
Cresylic acid, 95 per cent, 53 pounds, at $0.77 per gallon	4.74
Linseed oil, 28 pounds, at $2.20 per gallon	8.00
Soy-bean oil, 28 pounds at $0.20 a pound	5.60
Rosin, 25 pounds, at $17.50 per 280 pounds	1.56
Soy-bean oil, 7 pounds	1.40
Cost of 100 pounds of saponified cresol solution made with—	
Linseed oil, 28 per cent	12.87
Soy-bean oil, 28 per cent	10.47
Rosin, 25 per cent, soy-bean oil, 7 per cent	7.83

Saponified cresol solution made with linseed oil (28 per cent) costs 64.5 per cent more than that made with rosin (25 per cent) soy-bean oil (7 per cent). Saponified cresol solution made with soy-bean oil (28 per cent) costs 33.7 per cent more than that made with rosin (25 per cent), soy-bean oil (7 per cent). Saponified cresol solution made with oleic acid, fish oil (menhaden), or soy-bean oil fatty acids, would cost more than the rosin (25 per cent), soy-bean oil (7 per cent).

Bacteriological tests,[3] using a modified Rideal-Walker method, were made of saponified cresol solutions containing cresol (50 per cent) and different soaps equivalent to 28 per cent linseed oil. No. 10 was made with rosin soap; No. 11, linseed-oil soap; No. 12, soy-bean-oil soap; No. 13, fish-oil soap; No. 14, rosin soap ¾, soy-bean-oil

[1] See Lewkowitsch, Chemical technology and analysis of oils, fats and waxes, ed. 5, vol. 1, p. 346, and Bureau of Standards Circular 62, p. 6.

[2] Oil, Paint and Drug Reporter, July 21, 1919.

[3] These tests were conducted by Dr. F. W. Tilley, of the Bureau of Animal Industry. The modification consisted in the use of an unadjusted culture medium prepared as recommended by the committee of the A. P. H. A., on standard methods of examining disinfectants, in the American Journal of Public Health, July, 1918.

soap $\frac{1}{4}$; and No. 15, rosin soap $\frac{3}{4}$, fish-oil soap $\frac{1}{4}$. Just enough sodium hydroxid was used to saponify the rosin and oil. All these saponified cresol solutions were made with the same sample of cresol.

No.	Phenol coefficient.
10	2.6
11	2.1
12	1.9
13	2.2
14	2.6
15	2.6

SUMMARY

Saponified cresol solutions have been made by using soap mixtures composed largely of rosin soap. These saponified cresol solutions have at least as great a disinfecting power as saponified cresol solutions made with linseed-oil soap, and are much cheaper. When diluted with water to a 3 per cent solution the saponified cresol solutions remain clear for varying periods, depending upon the amount of rosin soap present.

Clouding in cresol-rosin-soap solutions is due to precipitation of rosin.

Saponified cresol solutions containing soaps made from several different vegetable oils and fish oil did not vary greatly in disinfecting power.

Saponified cresol solutions containing rosin soap appear to have a somewhat higher disinfecting power than those containing only vegetable oil or fish oil.

ADDITIONAL COPIES
OF THIS PUBLICATION MAY BE PROCURED FROM
THE SUPERINTENDENT OF DOCUMENTS
GOVERNMENT PRINTING OFFICE
WASHINGTON, D. C.
AT
5 CENTS PER COPY
△